PATENT SPECIFICATION

1,174,313

DRAWINGS ATTACHED.

Inventor:—LEONARD CHILL.

1,174,313

Date of Application (No. 24251/67) and filing Complete Specification: 24 May, 1967.

Application made in United States of America (No. 552,523) on 24 May, 1966.

Complete Specification Published: 17 Dec., 1969.

Index at acceptance:—B5 B(2P3, 20A, 21F1, 24C2, 25D1, 26A1).

International Classification:—B 29 d 7/24.

COMPLETE SPECIFICATION.

Process for Uniaxially Orienting Polypropylene Films.

We, CHEVRON RESEARCH COMPANY, a corporation duly organized under the laws of the State of Delaware, United States of America, and having offices at 200 Bush Street, San Francisco, California, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a novel multistage process for uniaxially orienting polypropylene films between sets of rollers having different peripheral speeds.

Stereoregular polypropylene films may be oriented to give them more tensile strength in the orientation direction. One method for orienting such film is to stretch it between rollers having different peripheral speeds. By such means thin polypropylene films have been oriented uniaxially in a single step to draw ratios (ratio of peripheral speeds of rollers) up to 6:1. However, in many instances films having tensile strengths greater than those obtained by orienting to draw ratios of 6:1 or less are desirable or essential. Additional drawing may increase the tensile strength. But, if attempted in a single stretch between rolls, the additional work required to reach these higher ratios causes the film temperature to rise substantially above that desired for orienting. These high temperatures may cause film breaks, erratic drawing, width fluctuation and low increases in film strength. These deficiencies are especially prevalent at high film speeds (above about 40 feet per minute casting speed).

A unique multi-stage process has now been found by which polypropylene film may be oriented uniaxially to ultimate draw ratios in the range of from 6:1 to below fibrillation, usually about 7:1 to 13:1 under conditions which control film temperature and the path over which the film is drawn. By this novel method unfibrillated film of uniform thickness in the range of 0.3 to 3.0 mils having longitudinal tensile strengths as great as 75,000 psi and even higher may be made. The process offers the additional advantage of being adaptable to wide films and high film speeds.

This novel method for orienting stereoregular polypropylene film uniaxially in the longitudinal direction to draw ratios between 6:1 and fibrillation is done in two or more successive integral orientation stages, preferably two or three. For the higher draw ratios in this range it is desirable to use more than two stages.

The first stage involves drawing the film adiabatically over a short, linear draw-span to a draw ratio of 2:1 to 5:1. In the follow stage(s) the film is further drawn to an ultimate draw ratio in the range of from 6:1 to below fibrillation. However, the following stage(s) draw is done essentially isothermally over a longer, partially arcuate path—most of the work required to orient to these ultimate ratios being done in the following stage(s).

In the first stage cast film about 2 to 20 mils thick is passed between a nip roll and a feed roll. The feed roll is internally heated to from 200 and 280°F., preferably 260 to 280°F. Its peripheral speed is substantially equal to the casting speed—which is usually about 5 to 150 feet per minute. The contact time of the film and feed roll is sufficient to heat the film to within 10°F. of the roll. The film is then drawn between the feed roll and a draw roll. Drawing occurs over a short linear path defined by the

[*Price* 4*s*. 6*d*.]

minimum tangential gap between the feed roll and draw roll. For conventional sized rolls, i.e., less than about 20 inch diameter, this path will usually be 0.2 to 3 inches long on a linear basis, more usually 0.2 to 2.0 inches long. The peripheral speed of the draw roll is 2 to 5 times, preferably about 4 times, that of the feed roll. The draw roll temperature is normally kept at 50 to 200°F., preferably 140 to 180°F. From this draw roll the film may be passed directly to the next following draw stage.

In the following stage the film is drawn under conditions which provide close control of the film temperature. The film first contacts a heated roll having a temperature of from 260 to 320°F., preferably 260 to 305°F. Its peripheral speed is substantially equal to that of the first stage draw roll. Contact time is sufficient to heat the film to within at least 10°F. of the heated roll. The film then passes to a draw roll having a peripheral speed sufficiently greater than the heated roll to give the desired amount of drawing. When only two orientation stages are used this will be about 1.2 to 5.0 times, preferably 2 times, that of the heated roll. Drawing is effected over a partially arcuate path at least about 6 inches long on a linear basis. The path is defined by the arc over which the film stretches in contact with the heated roll and the minimum tangential gap between the heated roll and following stage draw roll. This arc is influenced by the heated roll temperature, diameter and surface as well as first-stage orientation conditions.

The following-stage draw roll temperature is not critical, but is usually maintained in the range of 50 to 200°F.

The major portion of the draw in the following stage occurs while the film is in contact with the heated roll. Consequently the heated roll functions initially as a heating means and then as a "heat sink" to absorb heat generated by the drawing. In this manner film temperature is controlled during the draw.

The polypropylene film which is oriented by this process may be extruded and cast by conventional means. For instance, polypropylene may be melted at about 450 to 550°F. in an extruder having a slot die. It may then be extruded through the die at the desired speed and thickness. The extruded film may be cast by passing it over one or more internally cooled casting rolls.

The cast polypropylene film may be fed directly to the first orientation stage of this invention. The contact time of the film and the first-stage feed roll will depend upon the length of the arc over which the film contacts the roll and the casting speed. At higher casting speeds it may be difficult to get enough heat transfer from the roll to the film to bring the film to within 10°F. of the feed roll temperature. Under such conditions it may be desirable to employ auxiliary heating means to partially heat the film before it reaches the heated roll. This may be accomplished by passing the film over preheat rolls or through a heated zone, such as an oven. Likewise it may be desirable to use preheat means in conjunction with the following stage heated roll.

The film is nipped against the feed roll in the first stage so that essentially all the drawing is done in the tangential gap between the feed roll and draw roll. Since this drawing occurs quickly over a very short air gap, it is essentially adiabatic. It was found that if the film is allowed to draw over the roll as well as the air gap, erratic "necking" takes place in all stages and frequent film breaks occur in the following stage.

At high casting speeds it may also be desirable to nip the film near its contact point with the first-stage draw roll and the following stage heated roll.

Pull rolls or a nip roll on the following stage draw roll may be used to provide the pull necessary to orient in the following stage.

The first stage feed and draw rolls and the following stage heated roll and draw roll are desirably made from smooth or polished materials having a high heat transfer capacity. Examples of such materials are polished plated steel, satin finished steel, and aluminus. Nip rolls are desirably made of smooth resilient materials such as rubber and plastic.

The first stage feed roll and following stage heated roll diameters will be correlated with film velocity and path to give sufficient contact time between the film and the respective rolls. The diameter of the following stage heated roll will also be correlated with the desired draw path. The following stage heated roll diameter will normally be at least about 3 in. The draw roll diameters of the first and subsequent stages are not critical.

Oil, steam, electricity or other acceptable means may be used to heat the first stage feed roll and following stage heated roll internally.

The polypropylene used in this process is usually at least 85% stereoregular and more usually at least 95% stereoregular. This polypropylene is essentially insoluble in refluxing heptane. The intrinsic viscosity of this polypropylene will generally be in the range of 2—4 dl./g., more usually about 2—3 dl./g. (measured in decalin at 135°C.). Comparably its melt flow rate is in the range of about 0.1—12g./10 minutes, more usually 2—10 g./10 minutes. (Measured by ASTM D 1238-57T, 2160 grams load at 230°C.)

The polypropylene used in this invention may also contain minor portions of other resins, U.V. stabilizers, antioxidants, heat stabilizers, lubricants, dye acceptors, pigments, dyes and the like.

The accompanying drawing may help to better understand the process of this invention. It is a schematic side view of means by which polypropylene may be oriented uniaxially in two stages according to this invention. The first orientation stage involves rolls 2, 3, 4 and 5; the second stage involves rolls 6, 7, 8, 9 and 10.

The cast film 1 is passed between nip roll 2 and heated feed roll 3. As the film moves in contact with roll 3 it picks up heat. The film is stretched in the tangential gap between roll 3 and draw roll 5. The ratio of the peripheral speeds of roll 5 to roll 3 is 2:1 to 5:1. Nip roll 4 is optional and will be used with high casting speed. By contact with roll 5 film 1 is cooled and stretching is terminated. From roll 5 film 1 passes to the second stage.

In the second stage the film is heated as it moves in contact with heated roll 7. Nip roll 6 is optional and will be used with high casting speeds. The film is drawn by the combined effect of draw roll 9 and nip roll 10. Draw roll 9 has a peripheral speed about 1.2 to 5 times that of roll 7. Nip roll 10 "anchors" the film against roll 9 so that it stretches the film. Nip roll 8 is optional and will be engaged for high speed operation. Drawing occurs over the periphery of roll 7 as well as between roll 7 and roll 9. Film velocity measurements indicate that the majority of stretching in the second stage occurs while the film is in contact with roll 7.

The following examples also serve to illustrate specific embodiments of this invention.

Example 1

Polypropylene (95% insoluble in refluxing heptane) containing 1 wt. % graphite was melted in a conventional extruder, extruded and cast at 10 feet per minute as a flat, thin film. The cast film was passed between a nip roll and a 3½ in. diameter feed roll heated internally to about 275°F. The film was drawn by a 3½ in. diameter draw roll to a draw ratio of about 3.2: 1. Essentially adiabatic drawing occurred over the 0.4 in. minimum tangential gap between the feed roll and draw roll.

From the draw roll the film was passed over a preheat roll internally heated to about 225°F. After the preheat the film was passed over a 3½ in. diameter roll heated internally to about 285°F. and having a peripheral speed of 32 feet per minute. The film was then drawn by a 3½ in. diameter draw roll having a peripheral speed of about 70 feet per minute—thus obtaining an ultimate draw ratio of about 7: 1. The major portion of the drawing occurred while the film was in contact with the heated roll. The minimum tangential gap between the heated roll and draw roll was 1.35 in.

The resulting unfibrillated oriented film had a mean thickness of 1.2 mills. Its tensile strength (measured by drawing to break) was 58,600 psi.

Example 2

Using the general method described in Example 1, cast polypropylene film was oriented uniaxially to an ultimate draw ratio of 7.6: 1. The peripheral speed of the second-stage draw roll was 76 feet per minute instead of 70 feet per minute.

The resulting unfibrillated film had a mean thickness of 1.3 mils and a tensile strength of 60,600 psi.

Example 3

Using the general method of Example 1 with polypropylene containing no graphite, cast film was oriented to an ultimate draw ratio of 8.0: 1. The peripheral speed of the first-stage draw roll was 40 feet per minute instead of 32 feet per minute and the second-stage draw roll peripheral speed was 80 feet per minute rather than 76 feet per minute.

The unfibrillated oriented film had a mean thickness of 1.0 mils and a tensile strength of 68,200 psi.

Example 4

By the general method of Example 1 cast polypropylene film was oriented uniaxially to an ultimate draw ratio of 9.5: 1. As compared to the peripheral speeds of Example 1, the first-stage draw roll was run at 40 feet per minute and the second-stage draw roll was run at 95 feet per minute. The unfibrillated oriented film had a mean thickness of 1.5 mils and a tensile strength of 71,500 psi.

Example 5

By the general method of Example 1, but with the first-stage feed roll and second-stage heated roll each replaced with 6 in. diameter rolls, polypropylene film cast at 30 feet per minute was oriented uniaxially to an ultimate draw ratio of 7.6: 1. As compared to the peripheral speeds of Example 1, the first-stage draw roll was run at 96 feet per minute and the second-stage draw roll was run at 228 feet per minute. Also, temperature of the preheat roll was 249°F. rather than 225°F.

Example 6

By the method of Example 5 polypropylene film cast at 50 feet per minute was oriented uniaxially to an ultimate draw ratio of 7.6: 1. As compared to the peripheral speeds of Example 5 the first-stage draw roll was run at 160 feet per minute and the

second-stage draw roll was run at 380 feet per minute.

EXAMPLE 7

Comparison runs were made in which polypropylene film was cast at 30 feet per minute and oriented in a single stage to draw ratios of 6: 1 and 6.5: 1. The apparatus used in this single stage orientation was essentially the same as that described as the second stage apparatus in Example 5. At a draw ratio of 6: 1 occasional lateral film breaks occurred. At 6.5: 1 frequent lateral film breaks occurred causing numerous interruptions in the film production.

EXAMPLE 8

Polypropylene film was cast at 18 feet per minute and oriented in three stages to an ultimate draw ratio of about 12: 1. The apparatus used in this three-stage orientation was that of Example 5 with the addition of another set of rolls identical to those used on the second stage of Example 5. The temperature of the third stage heated roll was 301°F. The peripheral speeds of the driven rolls on each stage were: first—83 feet per minute; second—153 feet per minute; third—224 feet per minute.

The unfibrillated films made by this invention may be used in the manufacture of cordage products, such as twines, textiles and other like products.

WHAT WE CLAIM IS: —

1. A process for orienting stereoregular polypropylene film uniaxially in the longitudinal direction to an ultimate draw ratio in the range of from 6: 1 to below fibrillation in two or more successive orientation stages, which comprises:

passing in a first orientation stage cast polypropylene film having a thickness of from 2.0 to 20 mils between a nip roll and a feed roll having a temperature of from 200 to 280°F., the contact time of the film and feed roll being sufficient to heat the film to within at least 10°F. of the feed roll temperature, and drawing the film through a 0.2 to 3 inches long linear path defined by the minimum tangential gap between the feed roll and a first draw roll having a peripheral speed of from 2 to 5 times that of the feed roll and a temperature of from 50 to 200°F., and

in at least one additional orientation stage drawing the film to said ultimate draw ratio over a heated roll and between said heated roll and a following stage draw roll so as to effect drawing over a partially arcuate path defined by the arc over which the film stretches in contact with the heated roll and the minimum tangential gap between the heated roll and the following stage draw roll, the major portion of said drawing occurring over said arc, the heated roll having a temperature of from 260 to 320°F. and a peripheral speed substantially equal to that of the draw roll of the preceding orientation stage, said following stage draw roll having a temperature of from 50 to 200°F. and a peripheral speed greater than that of the heated roll and the contact time of the film and heated roll being sufficient to heat the film to within at least 10°F. of the heated roll temperature.

2. Process according to Claim 1, wherein there is only one additional orientation stage and the peripheral speed of the draw roll in the additional orientation stage is from 1.2 to 5 times that of the heated roll.

3. Process according to Claim 2, wherein the peripheral speed of the first draw roll is about 4 times that of the feed roll and the peripheral speed of the draw roll of the additional orientation stage is about 2 times that of the heated roll.

4. Process according to Claim 1, 2 or 3, wherein the temperature of feed roll is between 260 and 280°F. and the temperature of the heated roll is between 260 and 305°F.

5. Process according to Claim 1, wherein there are two additional orientation stages.

6. Process for orienting stereoregular polypropylene film, substantially as hereinbefore described with reference to the accompanying drawing.

7. Process for orienting stereoregular polypropylene film, substantially as described in any one of the foregoing Examples 1 to 6 and 8.

8. Uniaxially oriented stereoregular polypropylene film, whenever produced by the process claimed in any preceding claim.

9. A cordage product manufactured from a polypropylene film as claimed in Claim 8.

HASELTINE, LAKE & CO.,
Chartered Patent Agents,
28 Southampton Buildings,
Chancery Lane, London, W.C.2.
Agents for the Applicants.

Printed for Her Majesty's Stationery Office by Burgess & Son (Abingdon), Ltd.—1969.
Published at The Patent Office, 25 Southampton Buildings, London, W.C.2,
from which copies may be obtained.

1174313 COMPLETE SPECIFICATION

1 SHEET *This drawing is a reproduction of the Original on a reduced scale*